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Sommerfeld coefficient of δ-Pu determined via a low-temperature specific heat Pu-Ce study

L. Havela,[1,*] P. Javorský,[1] A. B. Shick,[2] J. Kolorenč,[2] E. Colineau,[3] J. Rebizant,[3] F. Wastin,[3] J.-C. Griveau,[3] L. Jolly,[4]
G. Texier,[4] F. Delaunay,[4] and N. Baclet[4]
[1]*Department of Condensed Matter Physics, Faculty of Mathematics and Physics, Charles University, Ke Karlovu 5, 12116 Prague 2, The Czech Republic*
[2]*Institute of Physics, Academy of Sciences of the Czech Republic, Prague 8, The Czech Republic*
[3]*European Commission, Joint Reseach Centre, Institute for Transuranium Elements, European Commission, Postfach 2340, 76125 Karlsruhe, Germany*
[4]*CEA, DAM, Valduc, 21120 Is-sur-Tille, France*
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Low-temperature specific-heat experiments on δ-Pu stabilized by Ce give the value of the Sommerfeld coefficient γ in the close vicinity of 40 mJ/mol K^2. The most precise data set for Pu-6.1 at. % Ce yields $\gamma=(41.5\pm0.5)$ mJ/mol K^2 and the Debye temperature $\theta_D=(103.0\pm0.5$ K). As Ce is in a compressed α-Ce state, major contribution to the γ value comes from the Pu states. Theoretical calculations suggest that the $5f^6$ admixture in the $5f^5$ ground state is responsible for the high-γ value. Although the $5f$ states are not present at the Fermi level, low-energy excitations due to transitions of the $5f^6\rightarrow5f^5$ type contribute to the spectral density around the Fermi level.

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I. INTRODUCTION

The Sommerfeld coefficient γ of electronic specific heat is one of fundamental material bulk properties carrying direct information on the density of quasiparticle states at the Fermi level. Understanding the behavior of δ-Pu, which is presumably the most strongly correlated allotropic modification of Pu metal, is one of the key issues in electronic structure of actinides.[1] The volume-expanded phase, which should have been magnetic on the basis of electronic-structure calculations, has in reality no magnetic moments.[2] This was surprising in the context of considerably enhanced value of the γ coefficient, taken often as a proof of $5f$ band residing at the Fermi energy. Already first experiments[3] were suggestive of largely enhanced $\gamma=(53\pm10)$ mJ/mol K^2 for δ-Pu stabilized by several percent of Al, which was contrasting with more modest value for α-Pu.

Despite recent progress in experiment,[4] amounting to even higher $\gamma=(64\pm3)$ mJ/mol K^2 for δ-Pu (again stabilized by Al), a large uncertainty was still remaining due to unfortunate combination of radiation self-heating, preventing reaching sufficiently low temperatures, and soft crystal lattice with pronounced non-Debye-type vibrations, making the separation of the lattice specific heat difficult. Additional obstacle is the tendency to form the monoclinic phase α' in a low-temperature martensitic transition for Al-stabilized Pu. All these factors caused that the γ value of δ-Pu remained still only poorly defined.

Therefore, we used in our previous study[5] a reliable dopant, Am. The improved phase stability was accompanied by the chance to study the reaction of γ to a lattice expansion. The fact that such expansion does not lead to any further γ enhancement is a strong argument against a narrow $5f$ band present at the Fermi level in δ-Pu. Instead, calculations yielding the $5f$ states off the Fermi level[6–9] have been gaining a firm ground, often reproducing the nonmagnetic ground state as well as basic cohesion parameters. The relatively high γ is then found as a result of many-body physics, either of the Kondo type or due to intra-ion excitations related to possible valence fluctuations.

The disadvantage of the Am doping is the strong self-heating due to ^{241}Am, posing a limitation on the lowest achievable temperature. In particular, precise determination of the γ value was not possible because of the uncertainty in the C/T extrapolation for $T^2\rightarrow0$. This led to a large error bar of γ. The Pu-Am study[5] concluded that γ lies within the range 35–55 mJ/mol K^2 for the Pu-8% Am sample, which is a lower value than published before.[3,4] The important finding, that γ does not increase upon expansion, eliminated simple band theories, which would lead to additional narrowing of the $5f$ band residing at the Fermi level. At this stage, the precise determination of γ, which is important benchmark for various theoretical approaches, remains an important task.

In the present work, we used the opportunity provided by the doping with Ce, which also leads to a volume expansion (somewhat smaller than for Am)[10] without any additional self-heating. Although Ce could, in principle, introduce artifacts due to possible many-body effects related to the instability of $4f$ states, the volume considerations prove that Ce is effectively in a volume collapsed α-Ce state, which exhibits no low-T anomalies. It can be deduced that the volume of Ce ions diluted in Pu corresponds to α-Ce at the pressure of 1.5 GPa. Considering the contribution of few percent of Ce to the total γ, we can, for example, use the γ value reported for α-Ce, $\gamma_{Ce}=21$ mJ/mol K^2.[11] A higher γ in the compressed phase with strong Ce valence fluctuations would be very unlikely. This can be understood comparing the γ values of other Ce valence fluctuators as CePd$_3$ (38.6 mJ/mol K^2) (Ref. 12) or CeSn$_3$ (60 mJ/mol K^2),[13] which have in addition a contribution related to the non-f components. Much larger enhancement could be expected only in the case of Kondo effect related to Ce atoms, which is for the compressed Ce atoms highly unlikely. In such case, an upturn in

C/T should be observed at low temperatures, which is not, as shown below, the case.

II. EXPERIMENT AND RESULTS

Unlike Am, the Ce stabilization of the fcc structure is not without problem. Phase diagram shows the range 4–17 at. % Ce as stabilizing the fcc structure at room temperature. But as found before,[10] the concentration 4.6% Ce is at the borderline, showing presumably a transition to α' phase, seen in electrical resistivity, below room temperature. The concentration 6.1% Ce has no such transition and therefore the stability of the cubic structure down to the lowest temperatures is granted. The lattice parameter a increases monotonously with increasing Ce concentration from 464 pm (value extrapolated to 0% Ce concentration), to 468 pm for 17% Ce. The increase is weaker (approximately twice) than for the Am doping, which yields $a \sim 468$ pm for 8% Am.

For our study, we chose the samples with the composition ^{239}Pu-4.6 at. % Ce, 6.1 at. % Ce, and 8.1 at. % Ce used in the previous work.[10] They were originally produced by induction melting of large amount of Pu (500 g) together with appropriate amount of Ce at 1100 °C for 2 h in Ta crucible. Casting into graphite molds produced ingots, which were subsequently annealed at 460 °C for 200 h. Prior to the specific-heat experiment, the samples were annealed for 6 h at 360 °C to remove the radiation damage. Specific-heat measurements were performed within 11 months from the annealing. X-ray diffraction proved the proper cubic crystal structure. The lattice parameters $a = (465.70 \pm 0.05)$ pm for Pu-6.1 at. % Ce and (466.40 ± 0.05) pm for Pu-8.1 at. % Ce should be compared to (467.60 ± 0.05) pm for Pu-8 at. % Am used in the previous study.[5]

For comparison, also the nonannealed sample with 6.1 at. % Ce was included. This sample was about a decade old and such samples exhibit traces of the α' phase. All samples were first cleaned from surface oxidic layer, which was more conspicuous than, e.g., in the Pu-Am samples treated in the same way. For the specific-heat data, especially the Ce oxide with its pronounced peak in $C(T)$ below 10 K could bring some problems. After the cleaning, the samples were divided into pieces of different mass and coated by precisely defined amount of Stycast.2850 FT.

The measurements were performed using the relaxation method[14] by means of the Quantum Design PPMS equipment. The temperature relaxations were analyzed using the 2τ method. The Stycast coating does not affect the heat conduction to the sample significantly, as proved by numerous studies.[15]

Generally, bigger samples (several milligram) provide better precision of absolute specific-heat determination; smaller samples (less than 1 mg) were used to achieve lowest temperatures. Therefore, two different measurements exist for each sample. Prior to every specific-heat measurement, addenda (sample substrate with Apiezon grease used to optimize the thermal contact) was measured under the same conditions. In the analysis process, addenda and the specific heat of the Stycast was subtracted. Standard correction for self-heating was performed but its impact is so low that it cannot be practically distinguished on the scale of the figures below.

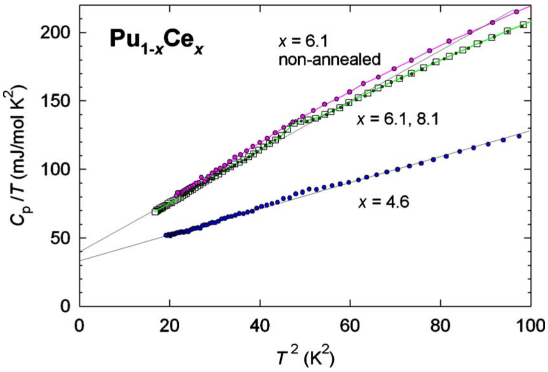

FIG. 1. (Color online) Low-temperature detail of the specific heat of Pu-Ce samples in the C_p/T vs T^2 representation. The plot contains raw data obtained on bigger samples (≈ 3 mg), which have a larger absolute accuracy. The data for Pu-6.1% Ce (open squares) and 8.1% Ce (small dots with full line, green in color version) practically coincide. The values for nonannealed sample Pu-6.1% Ce (magenta in the color version) are somewhat higher. The values for Pu-4.6% Ce (blue in color version) are much lower. The straight lines are linear extrapolation to $T=0$.

The overall temperature dependence $C_p(T)$ (not shown here) agrees well for all samples with the data published previously on Pu-Am alloys, with fast saturation to the value approximately 30 J/mol K. In the case of the lowest Ce concentration, 4.6%, the initial increase is slightly slower and approach to saturation is somewhat shifted toward higher T, indicating an increase in the Debye temperature Θ_D. The same is revealed with much better resolution in the C_p/T vs T^2 representation of the low-temperature part (Fig. 1). In the low-temperature limit of the Debye model, one assumes $C_p \approx C_v = \gamma T + \beta T^3$ so one can expect that a straight line is followed by C_p/T vs T^2, which allows to determine the γ value by extrapolation to $T \rightarrow 0$, and the slope β gives the Debye temperature θ_D ($\beta = 1944/\theta_D^3$ for C_p in J/mol K). The four data sets presented in Fig. 1 form two different groups. The lower slope (higher θ_D) and lower γ value of the 4.6% Ce sample are clearly related to the mixed-phase $(\alpha' + \delta)$ character. The admixture of α' modifies both parameters in the expected direction ($\gamma = 17$ mJ/mol K^2 and $\theta_D = 153$ K were reported for α-Pu).[4] The data for the samples with 6.1% and 8.1% Ce practically coincide on the given scale. The nonannealed sample with 6.1% Ce is also very similar, having the C_p values marginally higher but aiming at approximately same γ value of 40 mJ/mol K^2. This proves that the actual aging of the annealed samples, being shorter than 1 year, cannot have any noticeable impact on the γ value.

A closer inspection also reveals a weak anomaly in the vicinity of $T^2 = 50$ K^2. It is most likely related to a small amount of a Ce-based sesquioxide. Pure Ce_2O_3 has a very pronounced specific-heat anomaly due to antiferromagnetic ordering, which sets in at 8.5 K.[16] The antiferromagnetic origin is corroborated by the fact that the anomaly is gradually shifted down and removed in magnetic fields on the scale of several tesla. For further analysis of the specific heat,

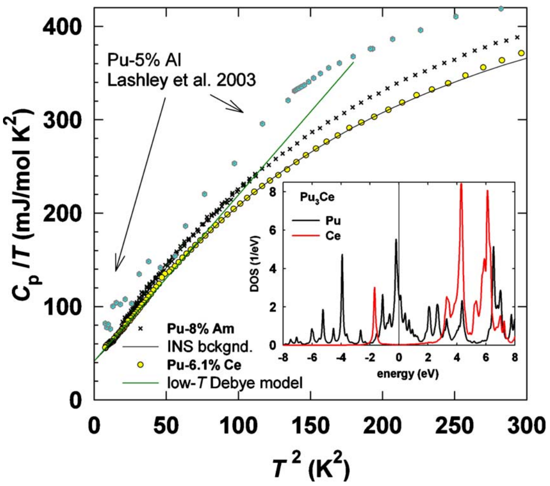

FIG. 2. (Color online) Comparison of the low-temperature specific heat in the C_p/T vs T^2 representation for Pu-6.1% Ce (circles, yellow in color version), with Pu-8% Am (crosses) from Ref. 5 and Pu-5% Al (more scattered circles on the top, cyan in color version) from Ref. 4. The full line represents the lattice contribution constructed from INS data in Ref. 4, superimposed with $\gamma = 41.5$ mJ/mol K^2. The straight line is the low-T limit of the Debye contribution with $\theta_D = 103$ K and $\gamma = 41.5$ mJ/mol K^2. The inset shows the spectral f densities for of the Pu (black) and Ce (red in online version) atoms in the Pu$_3$Ce alloy calculated by means of the LDA+HIA method (Ref. 17).

the temperature interval around the anomaly was excluded.

Figure 2 compares the C_p/T data of Pu-6.1% Ce with earlier data published on Pu-8% Am (Ref. 5) as well as with the Pu-5% Al data of Ref. 4. While the last data set has more scatter and points to the published higher $\gamma = (64 \pm 3)$ mJ/mol K^2, the data for Am-doped Pu, which have better statistic, miss the low-T end due to the stronger self-heating. The present data for Pu-6.1% Ce extend down enough to low temperatures so as to provide a sufficient linear part, needed for a precise extrapolation. The values of C_p are somewhat lower than for the Am doping but the slope of C_p/T vs T^2 is higher for the Am doping (corresponding to $\theta_D = 95$ K comparing with $\theta_D = 103$ K for Pu-6.1% Ce), so the respective γ values will be approximately equal. The low θ_D values explain why the deviation from linearity starts already below $T = 10$ K.

The lattice contribution to the specific heat behind the Debye approximation can be modeled on the basis of inelastic neutron scattering (INS). We followed the procedure introduced in Ref. 4, using the INS spectra obtained on Al-stabilized δ-Pu. Although we tried to use a similar type of analysis in Ref. 5 for analysis of Am-doped samples, Fig. 2 shows a small but systematic deviation of the Pu-8% Am data, which have somewhat too high slope in the low-T part. This simply means that the θ_D value for the Am-doped Pu is somewhat lower, probably due to the lattice expansion. This misfit is emphasized when noticing a perfect fit of the INS background with the present Pu-6.1% Ce data, which have the expansion smaller and the elastic properties of the lattice are likely to be much closer to the Al-doped Pu. This perfect fit documents the quality of the experimental data because the only adjustable parameter is the electronic contribution taken as γT. Figure 2 also illustrates how the INS background approaches the Debye low-T limit with $\theta_D = 103$ K. This fact allows to use a simple linear regression for the precise γ determination, if we restrict ourselves to the temperature range below the occurrence of the weak extrinsic anomaly.

TABLE I. Summary of analysis of the low-temperature ($T^2 < 40$ K^2) specific heat, providing the Debye temperature θ_D, Sommerfeld coefficient γ, and its values recalculated per mole Pu, using the γ value of α-Ce to approximate the small Ce contribution.

	a (pm)	θ_D (K)	γ (mJ/mol K^2)	γ_{Pu} (mJ/mol Pu K^2)
Pu-6.1% Ce	465.7	103.0 ± 0.5	41.5 ± 0.5	42.8 ± 0.5 [a]
Pu-8.1% Ce	466.4	102 ± 1	38 ± 1	40 ± 1
Pu-8% Am	467.6	95 ± 1	35 ± 2	38 ± 2

[a] If the specific-heat contribution from Ce would be taken as 0 (limit case) $\gamma = 44.2$ mJ/mol Pu K^2 would be obtained.

The results of such analyses for both concentrations of Ce are included in Table I, together with the data on Pu-8% Am analyzed in the same way. The errors include both the error of experimental data including the uncertainty of respective sample masses and the uncertainty of the fitting parameters. The larger errors for Pu-8.1% Ce comparing with Pu-6.1% Ce are partly due to the shorter T range covered.

III. ELECTRONIC-STRUCTURE CALCULATIONS

In order to gain understanding of microscopic origin of the γ coefficient in PuCe alloys, we employed electronic-structure calculations. They are based on the local-density approximation+Hubbard I approximation (LDA+HIA) method, including self-consistency over the charge density, implemented in the full-potential linearized augmented plane-wave basis.[17] To estimate the tendencies of γ variations and for the sake of simplicity, we considered the Pu$_3$Ce alloy, compared with Pu$_3$Am in the same fcc supercell. The Coulomb parameter $U_{Ce} = 6.1$ eV, exchange $J_{Ce} = 0.7$ eV, $U_{Am} = 4.5$ eV, exchange $J_{Am} = 0.67$ eV, and $U_{Pu} = 4.5$ eV, $J_{Pu} = 0.64$ eV were used. The experimental lattice constant of Pu$_3$Am (473.3 pm) was used in the calculations.

The γ coefficient was obtained as $\gamma = \frac{\pi^2}{3} k_B^2 \, \mathrm{Tr}[N(E_F)(1 - d\,\mathrm{Im}[\Sigma(i\omega)]/d\omega|_{\omega=0}]$, where $N(E_F)$ is the density of states (DOS) matrix at the Fermi energy and $\Sigma(\omega)$ is the Hubbard-I self-energy. The results are included in Table II. In addition

TABLE II. The f-shell occupations and f-shell contributions into the Sommerfeld coefficient γ in Pu$_3$Ce and Pu$_3$Am alloys resulting from the charge-density self-consistent LDA+HIA.

	Pu-n_{5f}	γ_{Pu} (mJ/mol K^2)	Ce/Am-n_f	$\gamma_{Ce/Am}$ (mJ/mol K^2)
Pu$_3$Ce	5.24	24.0	0.76	0.21
Pu$_3$Am	5.24	26.0	6.00	0.41

to the given f contributions, there is $2-3$ mJ/mol K^2 contribution to γ from non-f states. There is only a very small contribution to γ coming from Ce (as well as Am) atoms, and the γ values are mostly due to the Pu-f states. They are a bit smaller than the γ value of 30.1 mJ/mol K^2 for δ-Pu obtained from the LDA+HIA calculations. This slight decrease is evidently caused by an increase in volume with alloying. We note that LDA+HIA yields somewhat higher γ value than 20.4 mJ/mol K^2 obtained from LDA+DMFT in Quantum Monte Carlo approach [18] and somewhat lower value than 37.9 mJ/mol K^2 from LDA+DMFT in Fluctuation-exchange approximation.[9] As mentioned in Ref. 18, the γ value in δ-Pu is very sensitive to small changes in a very steep spectral density in the close vicinity of Fermi edge.

The f-projected spectral DOS (fDOS) for Pu and Ce atoms in Pu$_3$Ce alloy is shown in inset of Fig. 2. The Pu-atom fDOS is very similar to the fDOS for δ-Pu,[17] which agrees well with valence-band photoelectron spectra.[19] It has to be stressed that the high (spectral) density of states at the Fermi level is not a ground-state property but comes from excitations due to $N \to N-1$ transitions. It does not correspond to a naïve view of having a "5f band" at E_F. In fact the ground state obtained in LDA+U mean-field calculations[20] has the narrow band of 5f states well separated below the Fermi level. That is why γ does not react by an increase to a lattice expansion, which would give a band narrowing. The excitations described by our calculations reflect mostly the intraionic excitations, i.e., there can be found a correspondence to atomic multiplets as seen in photoelectron spectroscopy. Naturally the low-energy excitations are also reflected in the γ value.

As to the Ce alloying, we conclude that it does not induce any significant changes in the Pu electronic structure. The Ce-atom fDOS resembles the positions of lower and upper Hubbard bands in the experimental photoemission and inverse photoemission data for α-Ce (Ref. 21) but fails to reproduce the quasiparticle peak at the Fermi level. This is due to the use of HIA, which does not include hybridization between f and non-f states. Hence, the present LDA+HIA calculations underestimate the Ce-atom contribution to the γ value. This can partly explain why the calculated γ is smaller than the experimental value for the PuCe alloys (Fig. 3).

IV. DISCUSSION AND CONCLUSIONS

The experimental γ value per mol Pu remains in all cases in the vicinity of 40 mJ/mol K^2, which underlines the fact that the 5f states are located out of the Fermi level in the ground state so nothing like band narrowing upon crystal expansion can play a role. The reason for enhanced γ (although it is lower in reality than values given previously, it remains record-high among elements) can be seen in excitations of the system. In particular, one type of excitations of the $5f^6 \to 5f^5$ type costs very little energy, as revealed in the calculated spectral density using the LDA+HIA (Refs. 7 and 22, and present work) and in agreement with the original results of LDA+DMFT,[8,9] showing a pronounced peak in the close vicinity of the Fermi energy. The peak associated with the $^6H_{5/2}$ final state is observed in valence-band photoelectron spectroscopy in conjunction with the $5f^6$ admixture in the $5f^5$ initial state.[7,8] The theoretical γ values derived explained a large part of the experimental values.

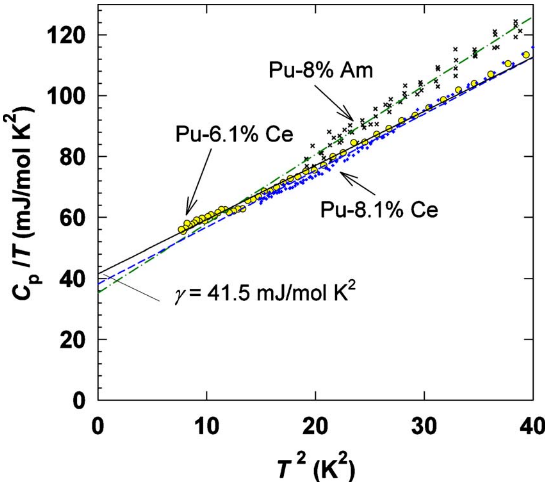

FIG. 3. (Color online) Low-temperature parts of data sets used for analysis of respective γ values. The full line represents the fit for Pu-6.1% Ce (data points circles, yellow in color version), the dashed line for Pu-8.1% Ce (data point small crosses, blue in color), and dashed-dotted for Pu-8% Am (green and big green crosses with a larger scatter for the data points).

With few exceptions, this picture is relevant for all metallic Pu-based systems so the enhanced γ values can be generally expected for Pu metal, Pu alloys, and compounds. Although an additional contribution due to heavy quasiparticles at E_F can be in general expected, too, one has to keep in mind that assumptions of, e.g., quantum criticality or other types of proximity to magnetism are not necessary in the present scenario. In reality, δ-Pu remains weak paramagnet even if expanded by Ce or Am doping, and γ stays high but more or less invariable, or weakly decreasing with the lattice expansion.

We can summarize that the Sommerfeld coefficient γ of δ-Pu is in the close vicinity of 40 mJ/mol K^2 and is not dramatically affected by doping and/or volume expansion.

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*Corresponding author.
FAX: +420-221911351; havela@mag.mff.cuni.cz

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